

Connecting via Winsock to STN

Welcome to STN International! Enter x:x

LOGINID:ssspta1202jxp

PASSWORD:

TERMINAL (ENTER 1, 2, 3, OR ?):2

* * * * * * * * * Welcome to STN International * * * * * * * * *

NEWS 1 Web Page for STN Seminar Schedule - N. America
NEWS 2 MAY 01 New CAS web site launched
NEWS 3 MAY 08 CA/CAplus Indian patent publication number format defined
NEWS 4 MAY 14 RDISCLOSURE on STN Easy enhanced with new search and display fields
NEWS 5 MAY 21 BIOSIS reloaded and enhanced with archival data
NEWS 6 MAY 21 TOXCENTER enhanced with BIOSIS reload
NEWS 7 MAY 21 CA/CAplus enhanced with additional kind codes for German patents
NEWS 8 MAY 22 CA/CAplus enhanced with IPC reclassification in Japanese patents
NEWS 9 JUN 27 CA/CAplus enhanced with pre-1967 CAS Registry Numbers
NEWS 10 JUN 29 STN Viewer now available
NEWS 11 JUN 29 STN Express, Version 8.2, now available
NEWS 12 JUL 02 LEMBASE coverage updated
NEWS 13 JUL 02 LMEDLINE coverage updated
NEWS 14 JUL 02 SCISEARCH enhanced with complete author names
NEWS 15 JUL 02 CHEMCATS accession numbers revised
NEWS 16 JUL 02 CA/CAplus enhanced with utility model patents from China
NEWS 17 JUL 16 CAplus enhanced with French and German abstracts
NEWS 18 JUL 18 CA/CAplus patent coverage enhanced
NEWS 19 JUL 26 USPATFULL/USPAT2 enhanced with IPC reclassification
NEWS 20 JUL 30 USGENE now available on STN
NEWS 21 AUG 06 CAS REGISTRY enhanced with new experimental property tags
NEWS 22 AUG 06 BEILSTEIN updated with new compounds
NEWS 23 AUG 06 FSTA enhanced with new thesaurus edition
NEWS 24 AUG 13 CA/CAplus enhanced with additional kind codes for granted patents
NEWS 25 AUG 20 CA/CAplus enhanced with CAS indexing in pre-1907 records
NEWS 26 AUG 27 Full-text patent databases enhanced with predefined patent family display formats from INPADOCDB
NEWS 27 AUG 27 USPATOLD now available on STN
NEWS 28 AUG 28 CAS REGISTRY enhanced with additional experimental spectral property data
NEWS 29 SEP 07 STN AnaVist, Version 2.0, now available with Derwent World Patents Index
NEWS 30 SEP 13 FORIS renamed to SOFIS
NEWS 31 SEP 13 INPADOCDB: New SDI frequency MONTHLY available now
NEWS EXPRESS 05 SEPTEMBER 2007: CURRENT WINDOWS VERSION IS V8.2, CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP), AND CURRENT DISCOVER FILE IS DATED 05 SEPTEMBER 2007.
NEWS HOURS STN Operating Hours Plus Help Desk Availability
NEWS LOGIN Welcome Banner and News Items
NEWS IPC8 For general information regarding STN implementation of IPC 8

Enter NEWS followed by the item number or name to see news on that specific topic.

All use of STN is subject to the provisions of the STN Customer agreement. Please note that this agreement limits use to scientific research. Use for software development or design or implementation of commercial gateways or other similar uses is prohibited and may result in loss of user privileges and other penalties.

FILE 'HOME' ENTERED AT 17:38:09 ON 16 SEP 2007

FILE 'REGISTRY' ENTERED AT 17:38:28 ON 16 SEP 2007
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2007 American Chemical Society (ACS)

Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 14 SEP 2007 HIGHEST RN 947298-73-9
DICTIONARY FILE UPDATES: 14 SEP 2007 HIGHEST RN 947298-73-9

New CAS Information Use Policies, enter **HELP USAGETERMS** for details.

TSCA INFORMATION NOW CURRENT THROUGH June 29, 2007

Please note that search-term pricing does apply when conducting SmartSELECT searches.

REGISTRY includes numerically searchable data for experimental and predicted properties as well as tags indicating availability of experimental property data in the original document. For information on property searching in REGISTRY, refer to:

<http://www.cas.org/support/stn/gen/stndoc/properties.html>

```
=>  
Uploading C:\Program Files\Stnexp\Queries\10550255.str
```

11 STRUCTURE UPLOADED

```
=> d l1
L1 HAS NO ANSWERS
L1           STR
*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***
```

Structure attributes must be viewed using STN Express query preparation.

```
=> s 11 sss full
FULL SEARCH INITIATED 17:39:18 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 103947 TO ITERATE
```

100.0% PROCESSED 103947 ITERATIONS 4893 ANSWERS
SEARCH TIME: 00.00.03

L2 4893 SEA SSS EUL 1.1

=>
Uploading C:\Program Files\Stnexp\Queries\10550255a.str

L3 STRUCTURE UPLOADED

=> d 13
L3 HAS NO ANSWERS
L3 STR
*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

Structure attributes must be viewed using STN Express query preparation.

=> s 13 sss full
FULL SEARCH INITIATED 17:42:10 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 281185 TO ITERATE

100.0% PROCESSED 281185 ITERATIONS 3150 ANSWERS
SEARCH TIME: 00.00.04

L4 3150 SEA SSS FUL L3

=> file caplus
COST IN U.S. DOLLARS SINCE FILE TOTAL
FULL ESTIMATED COST ENTRY SESSION
346.45 346.66

FILE 'CAPLUS' ENTERED AT 17:42:29 ON 16 SEP 2007
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2007 AMERICAN CHEMICAL SOCIETY (ACS)

Copyright of the articles to which records in this database refer is held by the publishers listed in the PUBLISHER (PB) field (available for records published or updated in Chemical Abstracts after December 26, 1996), unless otherwise indicated in the original publications. The CA Lexicon is the copyrighted intellectual property of the American Chemical Society and is provided to assist you in searching databases on STN. Any dissemination, distribution, copying, or storing of this information, without the prior written consent of CAS, is strictly prohibited.

FILE COVERS 1907 - 16 Sep 2007 VOL 147 ISS 13
FILE LAST UPDATED: 14 Sep 2007 (20070914/ED)

Effective October 17, 2005, revised CAS Information Use Policies apply.
They are available for your review at:

<http://www.cas.org/infopolicy.html>

=> s 12
L5 5501 L2

=> s 14
L6 7837 L4

=> s 15 and 16
L7 91 L5 AND L6

=> file reg
COST IN U.S. DOLLARS SINCE FILE TOTAL
ENTRY SESSION

FULL ESTIMATED COST

2.35

349.01

FILE 'REGISTRY' ENTERED AT 17:45:33 ON 16 SEP 2007
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2007 American Chemical Society (ACS)

Property values tagged with IC are from the ZIC/VINITI data file
provided by InfoChem.

STRUCTURE FILE UPDATES: 14 SEP 2007 HIGHEST RN 947298-73-9
DICTIONARY FILE UPDATES: 14 SEP 2007 HIGHEST RN 947298-73-9

New CAS Information Use Policies, enter HELP USAGETERMS for details.

TSCA INFORMATION NOW CURRENT THROUGH June 29, 2007

Please note that search-term pricing does apply when
conducting SmartSELECT searches.

REGISTRY includes numerically searchable data for experimental and
predicted properties as well as tags indicating availability of
experimental property data in the original document. For information
on property searching in REGISTRY, refer to:

<http://www.cas.org/support/stngen/stndoc/properties.html>

=>
Uploading C:\Program Files\Stnexp\Queries\10550255b.str

L8 STRUCTURE UPLOADED

=> s 18 sss full
FULL SEARCH INITIATED 17:46:09 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 28541 TO ITERATE

100.0% PROCESSED 28541 ITERATIONS 93 ANSWERS
SEARCH TIME: 00.00.01

L9 93 SEA SSS FUL L8

=> file caplus
COST IN U.S. DOLLARS SINCE FILE TOTAL
ENTRY SESSION
FULL ESTIMATED COST 172.10 521.11

FILE 'CAPLUS' ENTERED AT 17:46:21 ON 16 SEP 2007
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2007 AMERICAN CHEMICAL SOCIETY (ACS)

Copyright of the articles to which records in this database refer is
held by the publishers listed in the PUBLISHER (PB) field (available
for records published or updated in Chemical Abstracts after December
26, 1996), unless otherwise indicated in the original publications.
The CA Lexicon is the copyrighted intellectual property of the
American Chemical Society and is provided to assist you in searching
databases on STN. Any dissemination, distribution, copying, or storing
of this information, without the prior written consent of CAS, is
strictly prohibited.

FILE COVERS 1907 - 16 Sep 2007 VOL 147 ISS 13
FILE LAST UPDATED: 14 Sep 2007 (20070914/ED)

Effective October 17, 2005, revised CAS Information Use Policies apply.
They are available for your review at:

<http://www.cas.org/infopolicy.html>

=> s 19
L10 54 L9

=> d his

(FILE 'HOME' ENTERED AT 17:38:09 ON 16 SEP 2007)

FILE 'REGISTRY' ENTERED AT 17:38:28 ON 16 SEP 2007
L1 STRUCTURE uploaded
L2 4893 S L1 SSS FULL
L3 STRUCTURE uploaded
L4 3150 S L3 SSS FULL

FILE 'CAPLUS' ENTERED AT 17:42:29 ON 16 SEP 2007
L5 5501 S L2
L6 7837 S L4
L7 91 S L5 AND L6

FILE 'REGISTRY' ENTERED AT 17:45:33 ON 16 SEP 2007
L8 STRUCTURE uploaded
L9 93 S L8 SSS FULL

FILE 'CAPLUS' ENTERED AT 17:46:21 ON 16 SEP 2007
L10 54 S L9

=> s 17 and 110
L11 4 L7 AND L10

=> d 111 ibib ab hitstr tot

L11 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 2004:857685 CAPLUS
DOCUMENT NUMBER: 141:331639
TITLE: Synthesis of 3-substituted 4-aminobenzenesulfonamides, intermediates in benzophenone reverse transcriptase inhibitor preparation
INVENTOR(S): Martin, Michael Tolar
PATENT ASSIGNEE(S): Smithkline Beecham Corporation, USA
SOURCE: PCT Int. Appl., 17 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004087881	A2	20041014	WO 2004-US9375	20040326
WO 2004087881	A3	20041111		
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
RW:	BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI,			

SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

EP 1628953 A2 20060301 EP 2004-758431 20040326

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, CY, TR, BG, CZ, EE, HU, PL, SK

JP 2006521402 T 20060921 JP 2006-509365 20040326

US 2006183944 A1 20060817 US 2005-550255 20050922

PRIORITY APPLN. INFO.: US 2003-458144P P 20030327

WO 2004-US9375 W 20040326

OTHER SOURCE(S): CASREACT 141:331639; MARPAT 141:331639

AB The present invention is directed to processes for the synthesis of intermediates useful in the preparation of non-nucleoside reverse transcriptase inhibitors. Thus, 3-substituted 4-aminobenzenesulfonic acid is reacted with DMF and a chlorinating agent to form the N-protected sulfonyl chloride which is reacted with ammonia to form the protected sulfonamide. This compound is deprotected to prepare the 3-substituted 4-

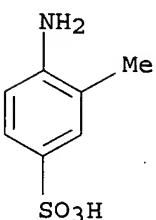
aminobenzenesulfonamide, which is useful in preparation of benzophenone derivs.

IT 98-33-9, 4-Amino-3-methylbenzenesulfonic acid

RL: RCT (Reactant); RACT (Reactant or reagent)
(synthesis of 3-substituted 4-aminobenzenesulfonamides, intermediates in benzophenone reverse transcriptase inhibitor preparation)

RN 98-33-9 CAPLUS

CN Benzenesulfonic acid, 4-amino-3-methyl- (CA INDEX NAME)

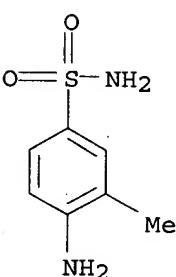


IT 53297-70-4P, 4-Amino-3-methylbenzenesulfonamide
770736-84-0P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(synthesis of 3-substituted 4-aminobenzenesulfonamides, intermediates in benzophenone reverse transcriptase inhibitor preparation)

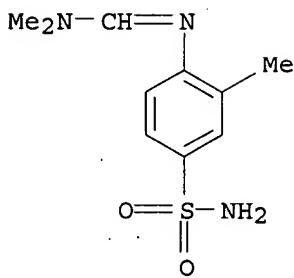
RN 53297-70-4 CAPLUS

CN Benzenesulfonamide, 4-amino-3-methyl- (CA INDEX NAME)

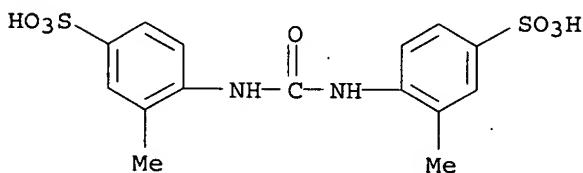


RN 770736-84-0 CAPLUS

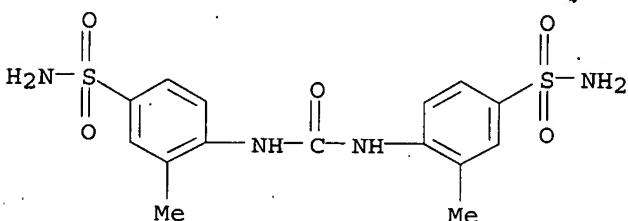
CN Benzenesulfonamide, 4-[(dimethylamino)methylene]amino-3-methyl- (9CI)
(CA INDEX NAME)



L11 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2007 ACS on STN
 ACCESSION NUMBER: 1973:97226 CAPLUS
 DOCUMENT NUMBER: 78:97226
 TITLE: Sulfonohydrazides and related compounds. XIII.
 AUTHOR(S): Cremlyn, Richard J. W.; Leonard, David; Motwani, Ramesh
 CORPORATE SOURCE: Dep. Chem. Sci., Hatfield Polytech., Hatfield, UK
 SOURCE: Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999) (1973), (5), 500-3
 CODEN: JCPRB4; ISSN: 0300-922X
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 AB p-NH₂CONHC₆H₄SO₂NHNH₂ (I) with ketones and aldehydes gave the hydrazones. (PhNH) ₂CO with ClSO₃H followed by N₂H₄ gave (p-NH₂NHO₂SC₆H₄NH) ₂CO, which reacted analogously to I. Similar chlorosulfonation of di-*o*-, *p*-, and *m*-tolylureas gave disulfonic acids. p-Succinimidobenzenesulfonyl chloride with NH₂NH₂.H₂O gave p-H₂NNHCO(CH₂)₂CONHC₆H₄SO₂NHNH₂ by ring cleavage.
 IT 40686-05-3P 40686-07-5P
 RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation of)
 RN 40686-05-3 CAPLUS
 CN Benzenesulfonic acid, 4,4'-(carbonyldiimino)bis[3-methyl- (9CI) (CA INDEX NAME)



RN 40686-07-5 CAPLUS
 CN Benzenesulfonamide, 4,4'-(carbonyldiimino)bis[3-methyl- (9CI) (CA INDEX NAME)



L11 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1968:114214 CAPLUS

DOCUMENT NUMBER: 68:114214

TITLE: Contrast media. III. Iodine derivatives of substituted sulfanilamides

AUTHOR(S): Radek, Otto; Kejha, Jiri; Nemecek, Oldrich; Kakac, Bohumil

CORPORATE SOURCE: Res. Inst. Pharm. Biochem., Prague, Czech.

SOURCE: Cesko-Slovenska Farmacie (1967), 16(1), 34-8

CODEN: CKFRAY; ISSN: 0009-0530

DOCUMENT TYPE: Journal

LANGUAGE: Czech

AB Sulfonamides serving as starting material for the title compds. were prepared by treatment of 4-acetamidobenzenesulfonyl chloride (I, R1 = Cl, R2 = R4 = H, R3 = NHAc) with various primary and secondary amines; some of them were new. The AcNH group was hydrolyzed with aqueous HCl and 4-aminobenzenesulfonamides were iodinated by ICl, KICl2, or NaICl2 to give the I shown in the table. Thus 22.0 g. I (R1 = NMe2, R2 = R4 = H, R3 = NH2) (Ia) was dissolved in 1200 ml. H2O and 60 ml. HCl at 60°, the solution cooled to 20°, and 110 ml. 2M KICl2 added. [TABLE OMITTED]

After a while an orange-yellow substance precipitated and the mixture was stirred

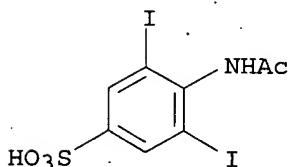
for 6 hrs. at 20° to give II. Ia (140 g.) was dissolved in 720 ml. H2O and 80 ml. HCl at 60°, the solution cooled to 40°, and 33 ml. 75% ICl in HCl added. The mixture was heated up to 90° during 1 hr. and kept for 5 hrs. to give III. I (R1 = NEt2, R2 = R4 = H, R3 = NH2) (20.52 g.) was dissolved in 1080 ml. H2O and 30 ml. HCl at 70°, cooled to 20°, and 100 ml. 2M NaICl2 was added. The mixture was stirred for 6 hrs. to give IV. Iodinated derivs. containing NHCONH2 could not be prepared and on attempted reaction of 4-ureidobenzenesulfonic acid or amide with ICl only 4-amino derivs. were obtained. The acylated 3,5-diiodo derivs. of sulfanilic acid are generally rather unstable; e.g. the acetyl group is lost from I (R1 = OH, R2 = R4 = I, R4 = NHAc) on attempted recrystn. III showed an antibacterial activity comparable with that of sulfadimidine, and compds. V and VI passed into gall bladder thus enabling its roentgenographic investigation.

IT 13192-25-1P 18229-71-5P 18229-72-6P

RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

RN 13192-25-1 CAPLUS

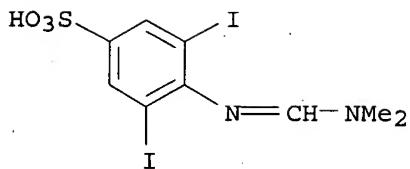
CN Benzenesulfonic acid, 4-(acetylamino)-3,5-diiodo-, monosodium salt (9CI)
(CA INDEX NAME)



● Na

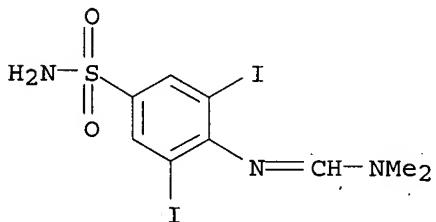
RN 18229-71-5 CAPLUS

CN Sulfanilic acid, N-[(dimethylamino)methylene]-3,5-diiodo- (8CI) (CA INDEX NAME)



RN 18229-72-6 CAPLUS

CN Sulfanilamide, N4-[(dimethylamino)methylene]-3,5-diido- (8CI) (CA INDEX NAME)



L11 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1960:129053 CAPLUS

DOCUMENT NUMBER: 54:129053

ORIGINAL REFERENCE NO.: 54:24766h-i,24767a-i,24768a-c

TITLE: Quinazolinone sulfonamides. A new class of diuretic agents

AUTHOR(S): Cohen, Elliott; Klarberg, Betty; Vaughan, James R., Jr.

CORPORATE SOURCE: Am. Cyanamid Co., Pearl River, NY

SOURCE: Journal of the American Chemical Society (1960), 82, 2731-5

DOCUMENT TYPE: CODEN: JACSAT; ISSN: 0002-7863

LANGUAGE: Journal

OTHER SOURCE(S): Unavailable

AB A series of 7-chloro-6-sulfamoyl-4(3H)-quinazolinones and

7-chloro-6-sulfamoyl-1,2,3,4-tetrahydro-4-quinazolinones were prepared and found to have a diuretic activity equal to or better than the benzothiadiazine 1,1-dioxides in exptl. animals. 4,2-Cl(H₂N)C₆H₃CO₂H (I) (17.2 g.) in 15 cc. 30% cold oleum treated dropwise with 11.6 g. ClSO₃H, the mixture heated 2.75 hrs. at 95-100° and 2 hrs. at 175-80° cooled to room temperature, poured onto 300 g. crushed ice, stirred, and filtered yielded 20.0 g. 5-SO₃H derivative (II) of I, pale yellow, m. above 360° (decomposition) (hot H₂O). II (20.0 g.), 200 cc. 98% HCO₂H, and 20.0 g. HCO₂Na heated 2 hrs. on the steam bath and evaporated, the residue triturated with a small amount of iced H₂O, refrigerated 1 hr., and filtered yielded 11.4 g. N-CHO derivative (III) of II, m. above 360° (aqueous dioxane). III (1.0 g.), 10 cc. POCl₃, and 1.5 g. PCl₅ heated 5 hrs. on the steam bath, cooled, and poured onto ice gave the 5-SO₂Cl analog of III. 5,2-ClMeC₆H₃NHAc (IV) (58 g.) and 100 cc. cold ClSO₃H containing 17 g. NaCl heated 2 hrs. on the steam bath, cooled, poured onto 500 g. ice, stirred, filtered, the residue added to 500 cc. concentrated NH₄OH, this heated 0.5 hr. with stirring to solution, kept 0.5 hr., filtered, the residue dissolved in aqueous NaOH, treated with C, and repptd. with concentrated HCl yielded

40 g. 4-SO₂NH₂ derivative (V) of IV, m. above 265°, and 6.0 g.

NaOH-insol. crystals; the NH₄OH filtrate concentrated gave 13 g. crystals, apparently the NH₄ salt of the 4-SO₃H derivative of V. V (40 g.) added in portions to 50 g. KMnO₄ in 500 cc. warm H₂O, the mixture heated 1.5 hrs. on the steam bath, cooled, filtered, the residue washed with 50 cc. dilute aqueous

NaOH, the combined filtrates treated with C, acidified, and cooled overnight gave 14 g. 5,2,4-Cl(HO₂C)(H₂NO₂S)C₆H₂NHAc (VI), m. 254-6°. VI (10 g.) dissolved with warming and stirring in 200 cc. EtOH and 200 cc. HCl, the mixture heated 0.5 hr. on the steam bath, cooled, concentrated in vacuo to near dryness, dissolved in 20 cc. N NaOH, clarified with C, acidified to Congo red, cooled overnight, and filtered gave 5.5 g. 5,2,4-Cl(HO₂C)(H₂NO₂S)C₆H₂NH₂ (VII), m. 275°; yields of 90% were obtained by saponification with 3N NaOH. VII (2 g.) and 2 g. EtCONH₂ heated 4 hrs. at 185-90°, the mixture stirred 1 hr. with cooling with 10% aqueous NaHCO₃, filtered, the residue dissolved in 20 cc. N NaOH, treated with C, and acidified with concentrated HCl gave 1 g.

7-chloro-2-ethyl-6-sulfamoyl-4(3H)-

quinazolinone (VIII), m. above 250°. VI (1.0 g.) and 1.0 g. H₂NCO₂Et heated 3 hrs. at 180-90°, cooled, treated with 10 cc. 10% aqueous NaHCO₃, the mixture stirred 1 hr., filtered, the residue dissolved in 5 cc. N NaOH, washed with EtOAc and Et₂O, treated with C, and repptd. with concentrated HCl yielded 2-Me homolog (IX) of VIII. VII, Bu₃N (or Et₃N), and ClCO₂Et (equivalent molar amts.) stirred 5-10 min. at -5 to -10°, the mixture treated with excess aqueous or liquid NH₃, stirred 10-30 min. in the cold and 10-30 min. on the steam bath, evaporated, the residue diluted with H₂O,

the mixture filtered, the filter residue stirred with aqueous NaHCO₃ to remove 50-70% VII, and the insol. solid recrystd. from EtOH yielded 20-30% 4,2,5-Cl(H₂N)-(H₂NO₂S)C₆H₂CONH₂ (X), m. 282-4° (decomposition); an increase in reaction temperature caused the formation of 2,4,5-Cl(H₂NCONH)(HO₂C)C₆H₂SO₂NH₂, m. 218°. VI converted to the glycol or Me ester and then amidated gave X. X (1.0 g.) in 100 cc. EtOH refluxed 1 hr. with 2 drops concentrated HCl and 0.70 cc. EtCH(OEt)₂, the mixture evaporated in

vacuo, and the residue triturated with 25 cc. H₂O yielded 0.90 g.

7-chloro-2-ethyl-6-sulfamoyl-1,2,3,4-tetrahydro-4-quinazolinone (XI), m. 250-2° (aqueous Me₂CO). NaCl (0.30 g.) and 3.0 cc. cold ClSO₃H treated dropwise with cooling with 1.0 g. 5,2-ClMeC₆H₃NH₂, the mixture heated 2 hrs. at 145°, cooled, poured on ice, filtered, the residue added to liquid NH₃, the solution evaporated at room temperature, the crude product (1.2 g.)

dissolved in 1.0N NaOH, the solution treated with HCl, and repptd. with HCl yielded 2,5,4-ClMe(H₂N)C₆H₂SO₂NH₂, m. 242-4° (aqueous EtOH). VII (1.0 g.) and 50 cc. (EtCO)₂O heated 3 hrs. on the steam bath, poured into 200 cc. iced H₂O, stirred, and filtered gave 0.65 g. 2,5,4-ClMe(EtCONH)C₆H₂SO₂NHCOEt·0.5H₂O, m. 237-8° (with effervescence). VII (5.0 g.) and 5 cc. HCONH₂ heated 3.5 hrs. at 170-5°, the mixture cooled, poured into 25 cc. cold H₂O, and triturated with MeOH yielded 1.8 g. crude product, which dissolved in 0.1N NaOH, treated with C, and repptd. with HCl gave 7-chloro-6-sulfamoyl-4(3H)-quinazolinone (XII), m. 310-15°. VII (1.0 g.) and 1.0 cc. HCONHMe heated 4 hrs. at 175-80° the mixture cooled, triturated with 5 cc. MeOH, filtered, the residue stirred with 5 cc. 10% aqueous NaHCO₃, dissolved in 0.1N NaOH, treated with C, and repptd. with HCl gave 0.25 g. 3-Me derivative (XIII) of XII. VII (0.5 g.) and 0.5 cc. AcNHMe heated 4 hrs. at 195-200°, the mixture cooled, triturated with MeOH, diluted with 5 cc. H₂O, refrigerated overnight, the solid (0.50 g.) stirred with 10% aqueous NaHCO₃, filtered, and repptd. in the usual manner yielded 2,3-di-Me derivative (XIV) of XII, m. 245°. VII (2.5 g.) and 2.5 g. iso-PrCONH₂ heated 3.5 hrs. at 190° dissolved in hot MeOH, concentrated slightly, diluted with H₂O, filtered, and the residue (1.0 g.) stirred with aqueous NaHCO₃ and purified in the usual manner yielded 0.20 g. 2-iso-Pr derivative (XV) of XII, needles, m. 290°. VII (0.50 g.) and 0.50 g. urea heated 3 hrs. at 180°, cooled, stirred with 10% aqueous NaHCO₃, filtered, and the residue (0.30 g.) purified in the usual manner gave 0.15 g. 7-chloro-2,4-dihydroxy-6-sulfamoylquinazoline (XVI), m. 275°. XII (2.0 g.) added with warming to 1.03 g. AlCl₃ in 250 cc. dry diglyme, the mixture treated dropwise with 1.4 g. NaBH₄ in 70 cc. dry diglyme, kept 1 hr. at 85°, cooled, treated slowly with 40 cc. H₂O, acidified, evaporated, and

the residue triturated with cold H₂O yielded 0.90 g. 1,2,3,4-tetrahydro derivative (XVII) of XII, m. 256-8°.. Similarly were prepared the tetrahydro derivs. of the following compds. (% yield and m.p. given): IX (XVIII), 60 (73% from X), 285°; XIII, 25, 257-9°; XIV, 47, 233-5°; XV, 60, 230°; Bu derivative of XVII.0.25H₂O, 70 (from X with BuCHO in diglyme), 219°. The average % change from the control in electrolyte excretion in dogs (orally) was determined for the following compds. (dose administered in mg./kg., % chloride and % potassium excretion during 24 hrs. given): chlorothiazide, 20, 266, 112; XII, 20, 261, 98; IX, 20, 223, 76; dihydrochlorothiazide (XIX), 1,201, 57; XIX, 5, 374, 122; XVIII, 1,150, 9; XVIII, 5, 360, 58; XVII, 1, 49, 22; XVII, 5, 212, 24; XI, 1,215, 101; XI, 5,291, 32.

IT 3086-91-7P, Anthranilic acid, 4-chloro-5-sulfamoyl-
 17560-53-1P, o-Aacetotoluidide, 5-chloro-4'-sulfamoyl-
 17560-54-2P, Anthranilic acid, N-acetyl-4-chloro-5-sulfamoyl-
 21501-33-7P, Anthranilic acid, N-carbamoyl-4-chloro-5-sulfamoyl-
 34121-17-0P, Benzamide, 2-amino-4-chloro-5-sulfamoyl-
 64174-54-5P, Anthranilic acid, 4-chloro-5-sulfo-
 72629-59-5P, m-Toluenesulfonamide, 4-amino-6-chloro-
 98557-08-5P, Sulfanilic acid, 2-chloro-5-(chloroformyl)-N-formyl-
 98557-46-1P, Anthranilic acid, 4-chloro-N-formyl-5-sulfo-
 99233-57-5P, m-Toluenesulfonic acid, 4-acetamido-6-chloro-
 RL: PREP (Preparation)
 (preparation of)

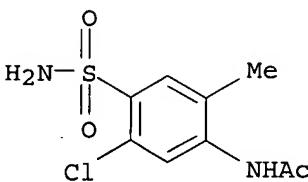
RN 3086-91-7 CAPLUS

CN Benzoic acid, 2-amino-5-(aminosulfonyl)-4-chloro- (9CI) (CA INDEX NAME)



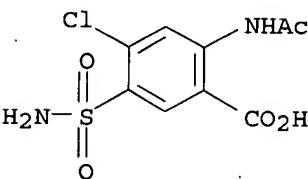
RN 17560-53-1 CAPLUS

CN Acetamide, N-[4-(aminosulfonyl)-5-chloro-2-methylphenyl]- (9CI) (CA INDEX NAME)

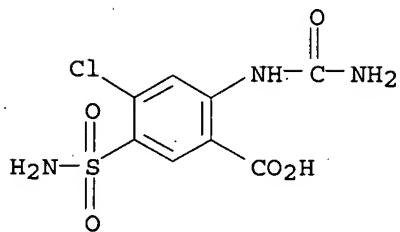


RN 17560-54-2 CAPLUS

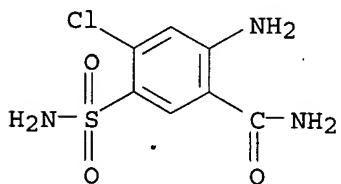
CN Benzoic acid, 2-(acetylamino)-5-(aminosulfonyl)-4-chloro- (9CI) (CA INDEX NAME)



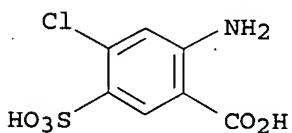
RN 21501-33-7 CAPLUS
CN Benzoic acid, 2-[(aminocarbonyl)amino]-5-(aminosulfonyl)-4-chloro- (9CI)
(CA INDEX NAME)



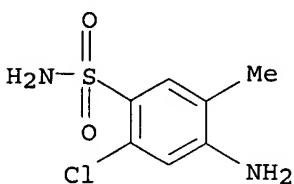
RN 34121-17-0 CAPLUS
CN Benzamide, 2-amino-5-(aminosulfonyl)-4-chloro- (9CI) (CA INDEX NAME)



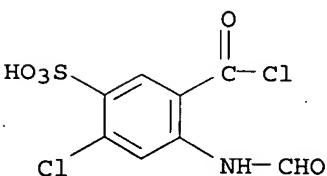
RN 64174-54-5 CAPLUS
CN Benzoic acid, 2-amino-4-chloro-5-sulfo- (9CI) (CA INDEX NAME)



RN 72629-59-5 CAPLUS
CN Benzenesulfonamide, 4-amino-2-chloro-5-methyl- (9CI) (CA INDEX NAME)

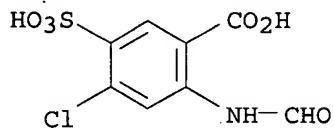


RN 98557-08-5 CAPLUS
CN Sulfanilic acid, 2-chloro-5-(chloroformyl)-N-formyl- (6CI) (CA INDEX NAME)



RN 98557-46-1 CAPLUS

CN Anthranilic acid, 4-chloro-N-formyl-5-sulfo- (6CI) (CA INDEX NAME)



RN 99233-57-5 CAPLUS

CN m-Toluenesulfonic acid, 4-acetamido-6-chloro- (6CI) (CA INDEX NAME)

